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Title: METHODS AND COMPOSITIONS FOR PREPARING SILICA AEROGELS

In the Claims

Please amend claims 1, 7, 13-14, 20-21, 23, 25-31, 36-43, 50, 63, 66-67, and 69-70 as follows:

- 1. (Currently Amended) A method to prepare a porous cross-linked metal oxide or silicon oxide based aerogel material, the method comprising:
- (a) first, forming a metal oxide or silicon oxide based sol-gel material to provide a preformed metal oxide or silicon oxide based sol-gel material; then,
- (a) (b) contacting [[a]] the preformed metal oxide or silicon oxide based sol-gel material with a cross-linking agent, the cross-linking agent comprising an organic compound, to provide a cross-linked metal oxide or silicon oxide based sol-gel material comprising organic cross-links; and then,
- (b) (c) drying the cross-linked metal oxide or silicon oxide based sol-gel material to form the porous cross-linked metal oxide or silicon oxide based aerogel material.
- 2. (Original) The method of claim 1, wherein the cross-linking agent is an isocvanate.
- 3 (Original) The method of claim 1, wherein the cross-linking agent comprises a diisocyanate, an acid anhydride, an acylchloride, a bis(acid anhydride), or a bis(acylchloride).
- 4. (Original) The method of claim 1, wherein the cross-linking agent comprises an attached group.
- 5. (Previously Presented) The method of claim 4, wherein the attached group is an absorbant, a catalyst, a fluorophore, a biomolecule, a redox active label, or a reactive group.
- 6 (Cancelled)
- 7. (Currently Amended) The method of claim 1 wherein the cross-linked sol-gel metal oxide or silicon oxide based aerogel material is a cross-linked silica-based-on-silica aerogel material.

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8. (Previously Presented) The method of claim 1, wherein the sol-gel material is

substantially filled with a solvent.

9. (Previously Presented) The method of claim 8, wherein the solvent is selected from the

group consisting of water, ketones, alcohols, esters, carbonates, lactones, hydrocarbons, and

mixtures thereof.

10. (Original) The method of claim 9, wherein the alcohol is methanol or ethanol.

11. (Original) The method of claim 9, wherein the ketone is acetone.

12. (Original) The method of claim 9, wherein the solvent is propylene carbonate, ethyl

acetate, or butyrolactone.

13. (Currently Amended) The method of claim 1, wherein the metal oxide or silicon oxide

based sol-gel material is a silica-based sol-gel material on silica.

14. (Currently Amended) The method of claim 13, wherein the silica-based sol-gel material

is prepared from silicon alkoxides via an acid or a base-catalyzed route.

15. (Original) The method of claim 1, wherein the cross-linking agent is in a solvent.

16. (Original) The method of claim 15, wherein the solvent is selected from the group

consisting of water, alcohols, ketones, esters, carbonates, lactones, and mixtures thereof.

17. (Original) The method of claim 16, wherein the alcohol is methanol or ethanol.

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combination thereof

18. (Original) The method of claim 2, wherein the isocyanate is a monoisocyanate, a diisocyanate, a triisocyanate, a tetraisocyanate, a polyisocyanate, an oligoisocyanate, or a

- 19. (Original) The method of claim 2, wherein the isocyanate is hexamethylene diisocyanate, poly(hexamethylene diisocyanate), toluene diisocyanate, diphenylmethane diisocyanate, an aliphatic polyisocyanate, triphenylmethyl triisocyanate, or a mixture thereof.
- (Currently Amended) The method of claim 1, wherein the <u>cross-linked metal oxide or</u> <u>silicon oxide based</u> sol-gel material is dried supercritically.
- (Currently Amended) The method of claim 20, wherein the <u>cross-linked metal oxide or silicon oxide based</u> sol-gel material is dried with liquid carbon dioxide.
- (Previously Presented) The method of claim 1, wherein the drying is conducted at ambient pressure.
- (Currently Amended) A porous <u>cross-linked metal oxide or silicon oxide based aerogel</u>
 material produced according to the method of claim 1.
- 24. (Cancelled).
- 25. (Currently Amended) A cross-linked metal oxide or silicon oxide based sol-gel material, wherein a metal oxide or silicon oxide based sol-gel material is formed to provide a preformed metal oxide or silicon oxide based sol-gel material, then a cross-linking agent comprising an organic compound is deposited on surfaces surrounding internal pores of [[a]] the preformed metal oxide or silicon oxide based sol-gel material so as to form a cross-linked metal oxide or silicon oxide based sol-gel material go granic cross-links.

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- 26. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent comprises at least about 2% by weight of the cross-linked metal oxide or silicon oxide based sol-gel material.
- 27. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent comprises at least about 5% by weight of the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material.
- 28. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent comprises at least about 10% by weight of the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material.
- 29. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent comprises at least about 30% by weight of the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material.
- 30. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent comprises at least about 50% by weight of the cross-linked <u>metal oxide or silicon oxide based sol-gel material</u>.
- 31. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent comprises at least about 80% by weight of the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material.
- 32. 35. (Cancelled)
- (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the sol-gel material is based on silica_based.

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37. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linking agent is an isocyanate.

- 38. (Currently Amended) The cross-linked metal oxide or silicon oxide based sol-gel material of claim 25, wherein the cross-linking agent is hexamethylene diisocyanate, poly(hexamethylene diisocyanate), toluene diisocyanate, diphenylmethane diisocyanate, an aliphatic polyisocyanate, triphenylmethyl triisocyanate, or a mixture thereof.
- 39. (Currently Amended) The cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material is stronger or more robust <u>more resistant to rupture under load</u> than a non-cross-linked <u>the preformed metal oxide or silicon oxide based</u> sol-gel material <u>prior to cross-linking with the cross-linking agent.</u>
- 40. (Currently Amended) <u>A cross-linked metal oxide or silicon oxide based aerogel material formed by drying of</u> the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 25, wherein the cross-linked sol-gel material is an aerogel <u>material and the cross-linked sol-gel material</u> does not collapse when in contact with a liquid that comprises water, an alcohol, an ether, a hydrocarbon, an ester, a ketone, a carboxylic acid, a phosphoric acid, or a liquefied gas.
- 41. (Currently Amended) The cross-linked sel-gel metal oxide or silicon oxide based aerogel material of claim 40, wherein the liquefied gas is nitrogen, argon, helium, hydrogen, or oxygen.
- (Currently Amended) The cross-linked sol-gel metal oxide or silicon oxide based acrogel
 material of claim 40, wherein the hydrocarbon is kerosene, gasoline, jet fuel, or rocket fuel.
- 43. (Currently Amended) A method to link an attached group to a <u>cross-linked metal oxide</u> or <u>silicon oxide based</u> sol-gel material comprising <u>the method of claim 1 wherein the</u> a) <u>contacting a preformed sol-gel material with a cross-linking agent comprising an organic compound that includes the attached group to <u>form a cross-linked sol-gel material; and</u></u>

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b) drying the cross-linked sol gel material.

- 44. (Previously Presented) A capacitor comprising the material of claim 23.
- 45. (Previously Presented) A dielectric comprising the material of claim 23.
- 46. (Previously Presented) An electrical circuit comprising the material of claim 23.
- (Previously Presented) A thermal insulating material comprising the material of claim
- 48. (Original) A tile, door, panel, shingle, shutter, beam, cooler, article of clothing, shoe, or boot comprising the thermal insulating material according to claim 47.
- 49. (Previously Presented) A structural material comprising the material of claim 23.
- 50. (Currently Amended) A method to dry the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material of claim 1 comprising:
- a) washing the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material, wherein the cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material is filled with a solvent, with a new solvent to exchange <u>pere-filling the</u> solvent of the cross-linked <u>metal oxide or silicon oxide</u> <u>based</u> sol-gel material with the new solvent; and
- b) drying the solvent-exchanged cross-linked <u>metal oxide or silicon oxide based</u> sol-gel material under non-supercritical conditions.
- 51. (Original) The method of claim 50, wherein the drying is conducted at ambient pressure.
- (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 4°C.

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 (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 20°C.

54. (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 40°C.

55. (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 60°C.

 (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 80°C.

57. (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 100°C.

 (Original) The method of claim 50, wherein the drying is conducted at a temperature that is at least about 200°C.

 (Original) The method of claim 50, wherein the drying is conducted at a temperature that is about or less than 300°C.

 (Original) The method of claim 50, wherein the drying is conducted at a temperature of about 40°C.

61. (Original) The method of claim 50, wherein the drying is by freeze-drying.

 (Previously Presented) The method of claim 50, wherein the new solvent is an organic solvent. Serial Number: 10/643,578
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- 63. (Currently Amended) The method of claim 62, wherein the organic solvent comprises a (C₁-C₂₀)alcohol, a (C₁-C₂₀)alkane, a (€₁-€₂₀) (C₂-C₂₀)cycloalkane, a (€₁-€₂₀) (C₂-C₂₀)alkene, a (€₁-€₂₀) (C₂-C₂₀)cycloalkene, a (€₁-€₂₀) (C₂-C₂₀)alkene substituted aryl, a (€₁-€₂₀) (C₂-C₂₀)alkene substituted aryl, a (€₁-€₂₀) (C₂-C₂₀)alkene substituted aryl.
- 64. (Original) The method of claim 62, wherein the organic solvent is a (C₅-C₁₀)alkane.
- 65. (Canceled).
- 66. (Currently Amended) The method of claim 1 wherein chemical functionality of surfaces surrounding pores of said metal oxide or silicon oxide based sol-gel material acts as a template for reaction with the cross-linking agent.
- (Currently Amended) The method of claim 1 wherein surfaces surrounding pores of the metal oxide or silicon oxide based sol-gel material act as a template for the cross-linking agent.
- 68. (Canceled).
- 69. (Currently Amended) The eross-linked metal oxide or silicon oxide based sol-gel material of claim 25 wherein a chemical functionality of the surfaces surrounding the pores of said metal oxide or silicon oxide based sol-gel material acts as a template for reaction with, accumulation of, or both, the cross-linking agent.
- 70. (Currently Amended) The eross-linked metal oxide or silicon oxide based sol-gel material of claim 25 wherein the surfaces surrounding the pores of the metal oxide or silicon oxide based sol-gel material act as a template for the cross-linking agent.